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ORIGINAL ARTICLE

Applicability comparison of different models for ammonium ion adsorption by multi-walled carbon nanotube

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Abstract The aim of the present research was to remove ammonium ions (NH_4^+) from aqueous solution using multi-walled carbon nanotubes (MWCNTs). The adsorption behavior of ammonium ions (NH_4^+) from aqueous solution by multi-walled carbon nanotubes (MWCNTs) was investigated as a function of some parameters such as contact time, initial ammonium ion concentration, and temperature. Adsorption equilibrium data for the removal of ammonium ions were examined by fitting the experimental data to various models. Six isotherm models were used to describe the isotherm experiment data. Langmuir, Freundlich, and Halsey isotherm models provided much better isotherm data fitting than the other isotherm models (Temkin, D–R, and Harkins–Jura). The Langmuir equation was found to best represent the equilibrium data for ammonium ion-MWCNT system. The average relative errors (ARE) value demonstrated that the Langmuir provided the best model for these data.

1. Introduction

Excess aqueous ammonia could contribute to the eutrophication of lakes and rivers, depletion of dissolved oxygen and toxicity to fish and other aquatic organisms. With comparing traditional methods (biological nitrification/denitrification, chemical coagulation, selective ion exchange, air stripping, chemical precipitation, etc.) (Rostron et al., 2001; Jorgensen and Weatherley, 2003; Bae et al., 2002) to removing aqueous ammonia from wastewater, the adsorption method is more economically feasible and environmentally friendly. Most studies on ammonium adsorbed from aqueous solution have been focused on mineral materials as adsorbents, such as zeolites (Huang et al., 2010; Zhao et al., 2010; Yusof et al., 2010; Lei et al., 2008) and limestone (Hussain et al., 2006). Plant materials are a kind of abundant reproducible natural resource and a type of waste. Therefore, they were studied extensively as low-cost adsorbents to remove a variety of pollutants from aqueous solution, e.g. heavy metals (Majumdar et al., 2010), dyes (Ho et al., 2005), nitrate (Orlando et al., 2002), phosphate (Eberhardt and Min, 2008), phenol (Cherifia et al., 2009) and ammonium ions (Liu et al., 2010a,b; Karadag et al., 2008; Baker and Fraij, 2010; Mara~nón et al., 2006; Karadag et al.,

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2006; Wahab et al., 2010). However, no information is available on the removal of aqueous ammonia by carbon nanotubes as an adsorbent from aqueous solution.

Carbon nanotubes (CNTs) as new adsorbents have gained increasing attention of many researchers, since Long and Yang (2001) first reported that CNTs were more efficient for the removal of dioxins than activated carbon. According to the grapheme layer, CNTs can be classified into single-walled CNTs (SWCNTs) and multi-walled CNTs (MWCNTs) (Trojanowicz, 2006). Due to their large specific surface area, small size, and hollow and layered structures, CNTs have been proven to possess great potential as superior adsorbents for removing many kinds of organic and inorganic contaminants including 1,2-dichlorobenzene (Peng et al., 2003), trihalomethanes (Lu et al., 2005), microcystins (Yan et al., 2006), fluoride (Li et al., 2001), lead (Li et al., 2002), nickel (Chen and Wang, 2006) and arsenate (Peng et al., 2005). Recently, Wu (2007) has investigated the adsorption equilibrium, kinetics and thermodynamic parameters of CNTs for the removal of reactive dye. Although the adsorption capacity is increased using CNTs as adsorbents, it might suffer from the inconvenience of tedious centrifugation separation process. To overcome this problem, Fugetsu et al. (2004) have encapsulated MWCNTs in Ba^{2+}-alginate matrix to constitute a cage and used it as the adsorbent for the elimination of ionic dyes.

In this account applicability of carbon nanotubes (CNTs) as an adsorbent to remove ammonium ions from aqueous solution is investigated and reported. The aim of the present study was to evaluate the potential and effectiveness of CNTs for NH_4^+ the removal from aqueous solution. It also provides information about the effect of various parameters such as contact time (t) and initial ammonium concentration (C) on the adsorption process. The adsorption capacity of the adsorbent has been studied by using the adsorption isotherm technique. Various isotherm equations were used to assess the best isotherm equation which represents the experimental data adequately and satisfactorily.

2. Materials and methods

2.1. Raw materials

MWCNTs (Armchair (6,6), Young's modulus (0.94T TPa), tensile strength (GPa 126.2T), purity, >95; diameter 1–2 nm; length, 5–30 nm; surface area, ~400 m^2/g; and manufacturing method, catalytic chemical vapor deposition (CVD) were purchased from NanoAmor Nanostructured & Amorphous Materials, Inc., USA. Doubly distilled water was used and all adsorbents were washed before using. Fig. 1 presents SEM image of carbon nanotubes. Ammonium chloride salt (NH_4Cl) (molecular weight, 53.16 g/mol) was supplied by Merck, Germany (maximum purity available). Doubly distilled deionized water (HPLC grade 99.99% purity) was obtained from Sigma Aldrich Co. (Germany).

2.2. Adsorption studies

For the purpose of the study of the adsorption process, firstly a 1000 mg/L stock solution of ammonium was prepared by dissolving 3.819 g ammonium chloride (NH_4Cl) in deionized water and adding water to 1000 mL. The solutions to be used were prepared by diluting the stock solution with deionized water when necessary.

Adsorption thermodynamic and kinetic experiments were conducted by using a 100 mL glass flask containing 0.05 g of the adsorbent and 10 mL of the ammonium ion solution with the initial concentration, 140 mg/L. The glass flask was sealed with a glass stopper. These samples were then mounted onto an Ultrasonic Bath for 5–45 min at various temperatures (288,

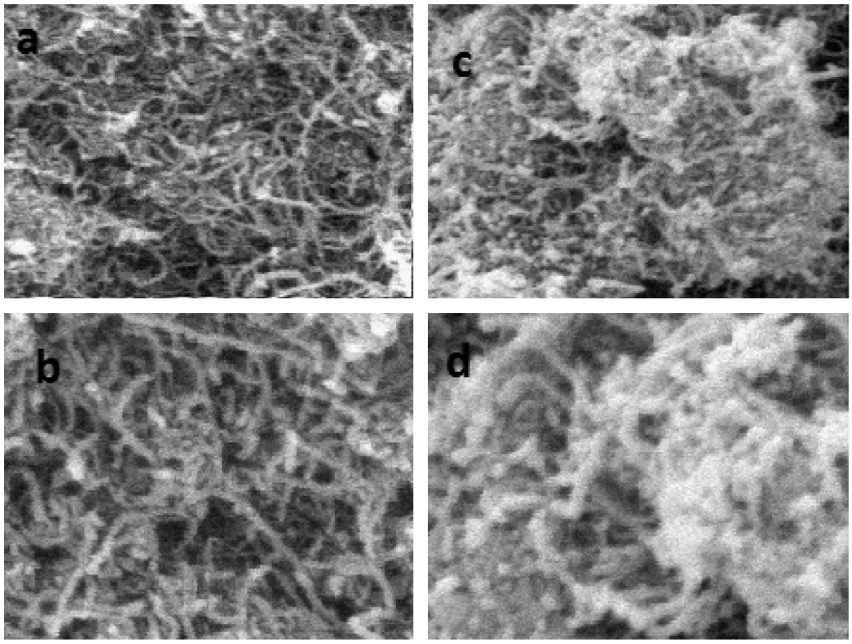

Figure 1 SEM images of carbon nanotubes (a) before adsorption 7500×, (b) before adsorption 15,000×, (c) after adsorption 7500×, and (d) after adsorption 15,000×.

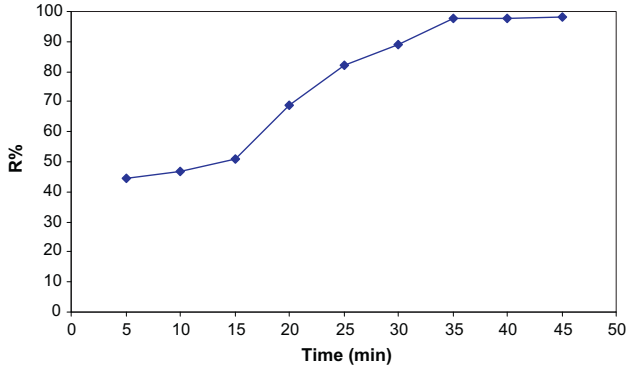

Figure 2 Effect of contact time on the percentage of ion adsorption onto CNT surface, initial concentration, 100 mg/L; adsorbent dosage, 50 mg/L and $T = 298$ K.

298, 308, 318 and 328 K). An Ultrasonic Bath (71020-DTH-E; Model 1510 DTH, 220V; EMS Company) was used to prevent the particles from aggregating and forming bulk. The samples were then centrifuged at 4500 rpm and the supernatant was then filtered through 0.2 μm filtered papers for subsequent analysis of ammonium concentration. The initial and final ammonium concentrations remaining in solutions were analyzed by a UV spectrophotometer (Varian-Cary100 Bio), monitoring the absorbance changes at a wavelength of maximum absorbance $\lambda = 400$ nm. The difference between the initial and the equilibrium ion concentration could determine the amount of ions adsorbed onto CNTs' surface. The amount of ammonium adsorbed was calculated using the following equation:

$$q_e = \frac{(C_0 - C_e)V}{m} \quad (1)$$

where C_0 and C_e (mg/L) are the initial ammonium liquid-phase concentration and its concentration at equilibrium, respectively. V is the volume of the solution (L) and m is the mass of adsorbent used (g). All experiments were performed in triplicate, and only the mean values have been reported. Ammoniacal nitrogen concentration was measured by Nesslerization method (APHA, 1998).

3. Results and discussion

3.1. Characterization of the CNT

The Fig. 1 showed morphologies of the CNT as adsorbent was obtained by scanning electron micrographs (SEM) (JEOL 6300F SEM). It was observed that ammonium ion was successfully adsorbed by CNT (Fig. 1, c & d). These results were indicated CNT as adsorbents for removal of ammonium ion from aqueous solution.

3.2. Effect of contact time

In order to determine the optimizing time required for the adsorption of NH_4^+ onto CNT, 10 mL, 100 mg/L solution was shaken with 50 mg CNT. The equilibrium studies were performed at the selected intervals of time ranging from 5 to 45 min. As shown in Fig. 2; after 35 min stirring of the solution, the removal efficiency did not increase. Therefore, the optimum value of stirring time was found to be 35 min. The change in the rate of NH_4^+ ions removal might be due to the fact that initially all adsorbent sites were vacant and the solute concentration is high. Afterward the NH_4^+ ions uptake rate by the CNT decreased significantly due to the decrease in vacant adsorption sites. On the basis of these results, a 35 min shaking period was selected for all further studies.

3.3. Effect of temperature on adsorption

The effect of temperature was studied at 288–328 K with 10 mL, 140 mg/L NH_4^+ solution and 0.05 g carbon nanotube.

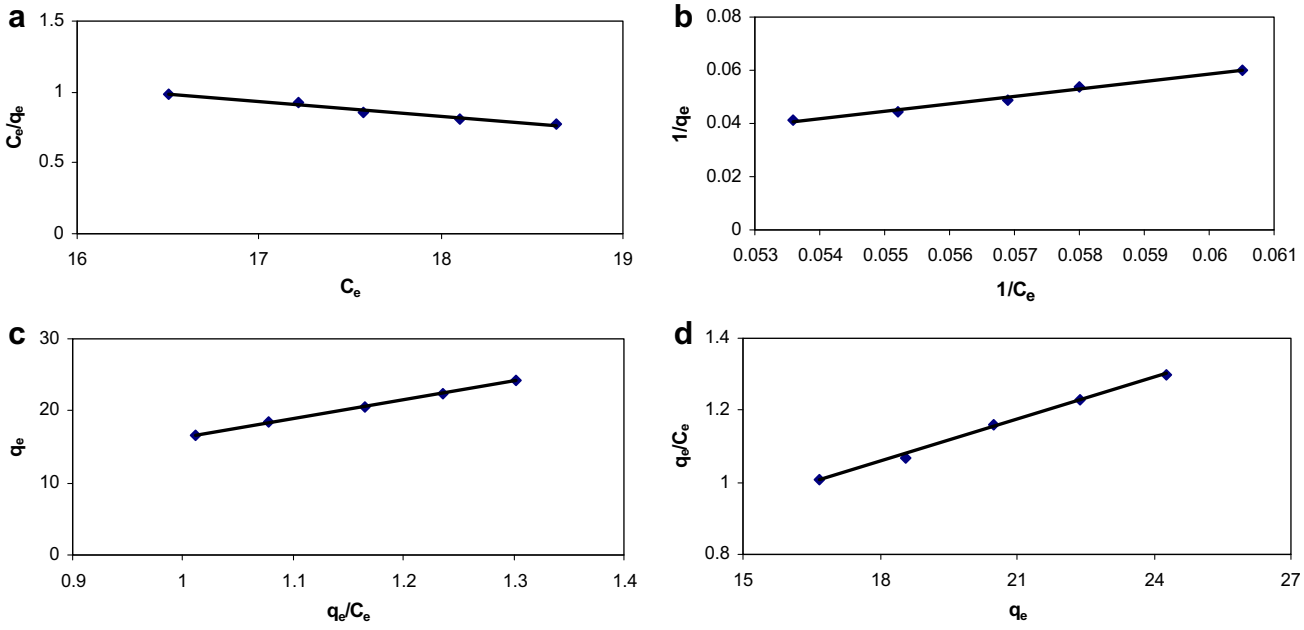

Figure 3 Langmuir isotherm of ammonium ions on CNTs surface (a) Type 1 Langmuir, (b) Type 2 Langmuir, (c) Type 3 Langmuir, and (d) Type 4 Langmuir.

The results are shown in Fig. 3; the adsorption capacity of NH_4^+ onto carbon nanotubes was found to decrease with increase in temperature, suggesting the process in this research has been exothermic. This may be due to a tendency for the NH_4^+ ions to escape from the solid phase to the bulk phase with an increase in the temperature of the solution.

3.4. Effect of initial ammonium concentration

The effect of initial ammonium concentration was studied at different initial ammonium ion concentrations in the range of 100–140 mg/L at 298 K with 0.05 g carbon nanotube at 35 min. Ammonium ion adsorption capacity increased with increasing initial ammonium ion concentration in solution. This can be attributed to the fact that higher concentrations result in a higher solute gradient, providing the necessary driving force for ammonium ions to be adsorbed onto surface of adsorbent.

3.5. Ammonium ion adsorption isotherms

The adsorption isotherm indicates how the adsorbate molecules are distributed between the liquid phase and the solid phase when the adsorption process reaches an equilibrium state. In other words, adsorption isotherm is basically important to describe how solutes interact with adsorbents, and it is also crucial in optimizing the usage of adsorbents. The analysis of the experimental data by fitting to different isotherm models is an important step in finding a suitable model that can be used for design purposes. Adsorption isotherm study was carried out by some isotherm models namely Langmuir, Freundlich, Temkin, Dubinin–Radushkevich (D–R), Harkins–Jura and Halsey. The applicability of the isotherm equation to the studied adsorption was compared by judging the correlation coefficients, R^2. The properties of the above mentioned isotherm models have been briefly described below.

3.5.1. Langmuir isotherm

One of the most common isotherm models which are widely used is the Langmuir model. It is observed that the Langmuir isotherms can be linearized to at least four different types. The Langmuir isotherm model can be expressed as

$$q_e = \frac{Q_m K C_e}{(1 + KC_e)} \tag{2}$$

where Q_m (mg/g) and K (L/mg) are Langmuir constants related to adsorption capacity and energy of adsorption, respectively (Hameed, 2009). The essential characteristics of a Langmuir isotherm can be expressed in terms of a dimensionless separation factor, R_L which describes the type of isotherm and is defined by (Webber and Chakkravorti, 1974)

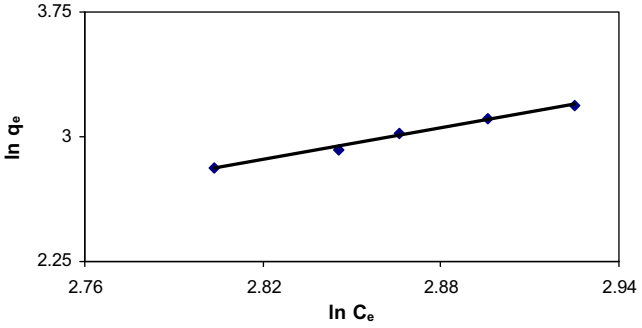

Figure 4 Freundlich adsorption isotherm of ammonium ions on CNTs surface.

$$R_L = \frac{1}{(1 + KC_0)} \tag{3}$$

If $R_L > 1$, unfavorable; $R_L = 1$, linear; $0 < R_L < 1$, favorable; $R_L = 0$, irreversible.

The R_L parameter lies between 0.15 and 0.20 which proves that the adsorption process is favorable and carbon nanotube is a potential adsorbent for the removal of ammonium ions from aqueous solution. The Langmuir plots for ammonium adsorption onto CNT were obtained and shown in Fig. 4. Table 1 summarizes the correlation coefficients of Langmuir isotherm.

3.5.2. Freundlich isotherm

Freundlich isotherm usually gives good curve fitting over a wider adsorbate concentration range, but its constants provide little physical insights into the adsorption mechanism. The values of these constants vary markedly with adsorbate concentration. It can be expressed as

$$q_e = K_F C_e^{1/n} \tag{4}$$

where K_F and $1/n$ are the Freundlich constants (Hameed, 2009). Similarly the Freundlich isotherm constants K_F and $1/n$ which are indicators of adsorption capacity and adsorption intensity, respectively, could be calculated from the plot of $\ln q_e$ versus $\ln C_e$. The results are provided in Fig. 5. The magnitude of $1/n$ quantifies the favorability of adsorption and the degree of heterogeneity on the surface of CNTs. If $1/n$ has a value less than unity suggesting favorable adsorption, adsorption capacity increases and new adsorption sites form Ozcan et al. (2004).

3.5.3. Temkin isotherm

Temkin isotherm model describes the behavior of adsorption systems on a heterogeneous surface, and can be expressed as

$$q_e = B \ln(AC_e) \tag{5}$$

where B is a constant related to adsorption heat and is equal to RT/b, A is the equilibrium binding constant corresponding to

Table 1 Langmuir isotherm parameters and ARE parameter for ammonium removal by CNTs surface.

Isotherm	Type	Equation	Q_m (mg/g)	K (L/mg)	R^2	R_L	ARE
Langmuir	Type 1	$C_e/q_e = 1/KQ_m + C_e/Q_m$	9.31	0.038	0.9798	0.15–0.20	0.20
	Type 2	$1/q_e = 1/Q_m + 1/KQ_mC_e$	9.18	0.039	0.9894		
	Type 3	$q_e = Q_m - q_e/KC_e$	9.20	0.039	0.998		
	Type 4	$q_e/C_e = KQ_m - Kq_e$	9.12	0.039	0.9975		

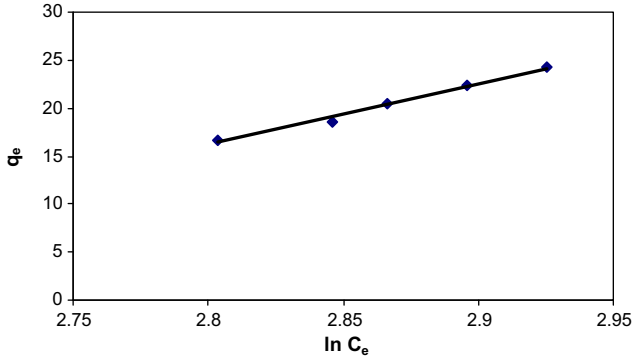

Figure 5 Temkin adsorption isotherm of ammonium ions on CNTs surface.

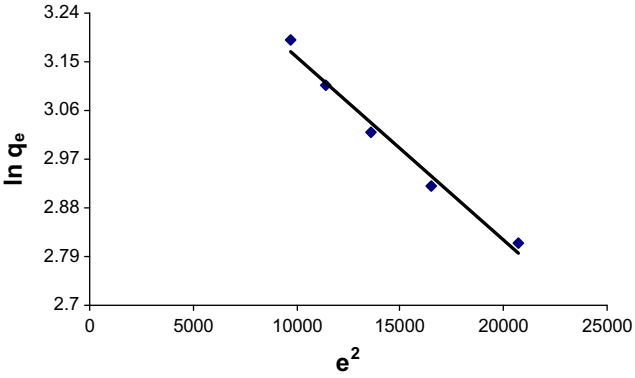

Figure 6 Dubinin–Radushkevich (D–R) adsorption isotherm of ammonium ions on CNTs surface.

maximum binding energy (Temkin and Pyzhev, 1940). As shown in Fig. 6 the plot of q_e versus $\ln C_e$ was used to determine isotherm constants.

3.5.4. Dubinin–Radushkevich isotherm

The Dubinin–Radushkevich (D–R) equation is given as

$$q_e = X'_m \exp(-K'\varepsilon^2) \tag{6}$$

where q_e is the amount of ammonium adsorbed per unit mass of CNTs. X'_m is adsorption capacity. K' is a constant related to adsorption energy (Hobson, 1969). ε is Polanyi potential and can be correlated as

$$\varepsilon = RT\ln\left(1+\frac{1}{C_e}\right) \tag{7}$$

R is the gas constant and T is adsorption temperature. K' gives the mean free energy E of sorption per molecule of sorbate when it is transferred to the surface of the solid from infinity in the solution and can be calculated using the following equation (Hameed, 2009):

$$E = \frac{1}{\sqrt{-2K'}} \tag{8}$$

The values of X'_m and K' were calculated from the intercept and slope of the $\ln q_e$ versus ε^2 plots as shown in Fig. 7.

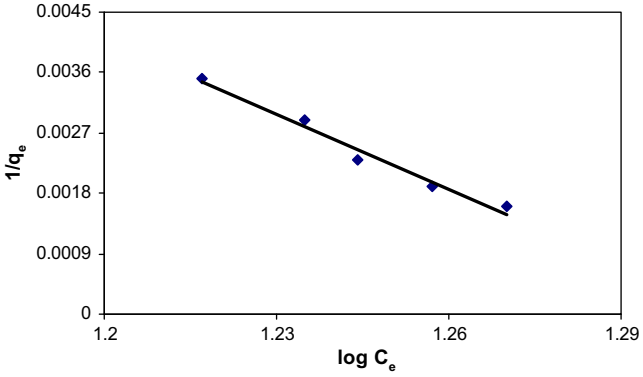

Figure 7 Harkins–Jura adsorption isotherm of ammonium ions on CNTs surface.

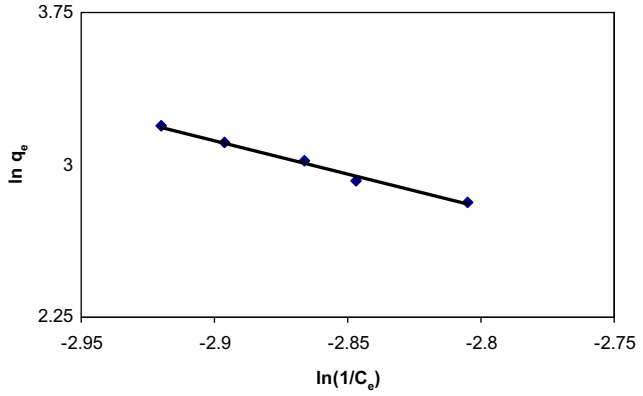

Figure 8 Halsey adsorption isotherm of ammonium ions on CNTs' surface.

3.5.5. Harkins–Jura and Halsey isotherms

The Harkins–Jura adsorption isotherm can be expressed as

$$\frac{1}{q_e^2} = \left(\frac{A}{B}\right) - \left(\frac{1}{A}\right)\log C_e \tag{9}$$

which A and B to multilayer adsorption that can be explained with the existence of a heterogeneous pore distribution.

The Halsey (Basar, 2006) adsorption isotherm can be given as

$$\ln q_e = \left[\left(\frac{1}{n}\right)\ln K\right] - \left(\frac{1}{n}\right)\ln C_e \tag{10}$$

This equation is suitable for multilayer adsorption. Especially, the fitting of this equation can be best used for heterotopous solids. $1/q_e$ versus $\log C_e$ Harkins–Jura isotherm (Fig. 7) and In $1/C_e$ Halsey adsorption isotherm are given in Fig. 8.

The experimental adsorption capacity and the predicted adsorption capacity by the isotherms and the corresponding isotherm parameters, along with the regression coefficients are listed and compared in Table 2.

3.5.6. Error function

The relative models' parameter values of those that express the surface properties and affinity of CNT are estimated by nonlinear regression analysis and are tabulated in Tables 1 and 2 with the average relative errors (ARE). The average relative

Table 2 D–R, Freundlich, Temkin, Harkins–Jura and Halsey and isotherm parameters and ARE parameter for ammonium removal by CNTs surface.

Dubinin–Radushkevich (D–R)	X'_m (mg/g)	K'	E (kJ/mol)	R^2	ARE
$\ln(q_e) = \ln(X'_m) - K'\varepsilon^2$	1.2518	−3E−05	4.09×10^{-6}	0.9841	0.29
Freundlich	K_F (mg/g)	$1/n$	R^2	ARE	
$\ln q_e = \ln K_F + 1/n \ln C_e$	1.79	0.1588	0.9918	0.21	
Temkin	A (L/mg)	B	R^2	ARE	
$q_e = B\ln(A) + B\ln(C_e)$	0.932	63.876	0.9889	0.27	
Harkins–Jura	A	B	R^2	ARE	
$\frac{1}{q_e^2} = \left(\frac{A}{B}\right) - \left(\frac{1}{A}\right)\log C_e$	26.80	1.30	0.9765	0.36	
Halsey	n	k	R^2	ARE	
$\ln q_e = \left[\left(\frac{1}{n}\right)\ln K\right] - \left(\frac{1}{n}\right)\ln C_e$	0.3012	0.67	0.9905	0.23	

errors between the experimental and predicted values are calculated using Eq. (11) (Ng et al., 2002) in which the subscripts 'exp' and 'calc' show the experimental and calculated values, respectively, and n is the number of measurements:

$$\frac{100}{n}\sum_{i=1}^{n}\left|\frac{q_{e,exp} - q_{e,calc}}{q_{e,exp}}\right| \tag{11}$$

The magnitude of average relative errors was the criteria for the selection of the most suitable isotherm model. On the basis of average relative errors in Table 1, the adsorption equilibrium data of ammonium ions fitted very well to the Langmuir model (type 3) in the concentration ranges studied for CNT. Moreover, the Freundlich and Halsey models were also found to satisfactorily describe the adsorption isotherms of ammonium ions over the whole concentration ranges studied in CNT systems with the relative error values less than 0.24%. However the other models of Temkin, D–R and Harkins–Jura exhibited a poor fit to the adsorption data of ammonium ions with an average relative error more than 0.26% (Foo and Hameed, 2010).

4. Conclusion

In this study, adsorption experiments were carried out for the adsorption of ammonium ions from aqueous solution using multi-walled carbon nanotube (CNT) surface as the adsorbent. The adsorption characteristics were examined at different contact time, initial concentration of ammonium ions, and temperature. Langmuir, Freundlich, Dubinin–Redushkevich (D–R), Temkin, Harkins–Jura, and Halsey equations were used to describe the adsorption of ammonium ions onto carbon nanotubes. Langmiur, Freundlich and Halsey models have high correlation coefficient than the other models in the studied concentration. R_L value from Langmuir isotherm model and $1/n$ from Freundlich isotherm model indicate that the removal of ammonium ions from the multi-walled carbon nanotube is favorable. The average relative errors (ARE) values of Langmuir, Freundlich, and Halsey models were 0.20%, 0.21%, and 0.23%, respectively.

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